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Research Article

Fabrication and Characterization of Synthesized Polysulfone/Polyvinylacetate Blend Membranes

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Abstract: In gas separation, the trade off limitations of polymeric membranes and economical consideration of inorganic membranes motivated the researchers to search for alternative approaches. Membranes with high selectivity and high permeability are required. Different modifications are in practice to improve the performance of membranes like mixed matrix, cross linking, grafting, polymer blending, making composite or hybrid membrane. It will be possible to develop polymeric blend membrane for separating high pressure gas streams at their processing pressure. This advantage could offer cost savings that may provide a new incentive for polymeric blend membranes. This result opens a new tool for studying gas separation by polymeric blend membranes. In this context blending of glassy and rubbery polymer that is Polysulfone and Polyvinyl acetate in dimethyl acetamide solvent, flat sheet membranes were developed with desirable properties. Membranes were synthesized with different blending ratios and the developed membranes were characterized by Fourier Transformed Infra-Red Spectroscopy (FTIR), Thermal Gravimetric Analysis (TGA), Field Emission Scanning Electron Microscopy (FESEM) and Gas Permeability to study the effect of blend ratio on different properties.

Keywords: Blending, CO_2/CH_4 gas separation, dimethylacetamide, polysulfone, polyvinyl acetate

INTRODUCTION

Membrane technology is very effective for separation of CO_2 from CH_4 and has been practiced for many years. It recommend number of advantages over other techniques which make it, to a certain extent appropriate for gas separation (Wee *et al*., 2008; Wind *et al*., 2004; Baker, 2004; Ismail and David, 2001; Sanders, 1988). There is also another key challenge in membrane separation process. Performance of polymeric membranes is limited under harsh operating conditions e.g., for CO_2 separation at high pressures there is a radical decrease in selectivity of membranes due to highly plasticization behavior of CO_2 (Khan *et al*., 2011).

Polymeric membranes perform their process by different mechanisms which are based upon the properties of membrane means physical and chemical structure. It interaction between membrane, components and nature of gas. Polymeric membranes can be classified on the basis on polymer material. Different studies regarding blend membrane are shown in Table 1.

Glassy polymers are hard, glass like structures and operate below glass transition temperature Tg, low permeability but a high selectivity like polysulfone and it has high glass transition temperature and many researchers are attracted to PSU to increase the permeability and selectivity (Ghosh and Banerjee, 2008; Mehdipour-Ataei and Amirshaghaghi, 2005; Zhang *et al*., 1999). PSU also considered to have good chemical and mechanical, thermal stability along with adequate gas permeability and selectivity (Van der Bruggen, 2009; Wang *et al*., 2000; Aitken *et al*., 1992).

Rubbery polymers are soft, flexible and operate above Tg, high permeability but a low selectivity like polyvinyl acetate.

There is a general tradeoff between permeability and selectivity for polymeric membranes with rubbery polymeric membranes with high permeability and low selectivity and vice versa for glassy polymeric membranes. In this context polymer blending means individual blend of glassy and rubbery offers time and cost effective method to develop materials with desirable properties and the upper bound line is cross. Therefore, effect of blending of a glassy and a rubbery polymer on the performance of polymeric membrane should be research so that a membrane with high selectivity and high permeability could be developed.

Thus in the current work, blending a glassy and rubbery polymer was carried out to prepare flat sheet dense polymeric blend membranes to see the effect of polymer blend ratio on morphology, polymer-polymer interaction and thermal stability of prepared membranes. Developed membranes were characterized by FESEM, FTIR, TGA and Gas Permeability, compared with individual polymers.

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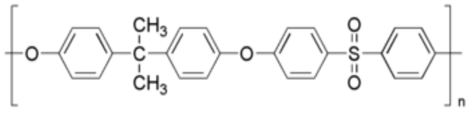
Table 1: Different studies regarding on blend membrane

Year	Polymers	System	Remarks
2002	PES/PI (glassy/glassy) coated with PDMS	Gas separation	Hollow fiber
2006	PU based PEI/PAI (glassy/glassy) blend	CO_2	
2006	PU/PDMS (rubbery/rubbery) cross-linked	Gas mixture	
2006	PVDF/PES (glassy/glassy)		Studied effect of polymer concentration, solvent and morphology
2009	PVAm/PVA with porous PES support	Facilitated CO_2 transport	Ultra-thin membrane with good strength, stability and permeability/selectivity
2008	PES/PI (glassy/glassy) blend MMM	O_2/N_2	
2010	PU/PDMS (rubbery/rubbery)	Methanol/toulene	Per vaporization
2010	PEG/PDMS	CO_2 separation	
2010	PU/CA (porous) (glassy/rubbery)	Micro filtration	
2010	PES/PI (glassy/glassy)	O_2/N_2	
2011	SPEEK/matrimid	CO_2 separation	Cross-linked for anti-plasticization
2011	PSF/PI (glassy/glassy)	CO_2/CH_4	Studied effect of solvents
2011	PEI/PVP	CO_2/CH_4, CO_2/N_2	Carbon hollow fiber membrane
2012	PES (PVP or PEG) with PDMS coating	Toulene/water	Per vaporization
2012	PU/PVAc with PEO/PPO	Gas mixture	Increased CO_2 permeability
2012	PIM-1/matrimid	CO_2/CH_4, CO_2/N_2	Increased selectivity

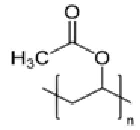
CA: Cellulose acetate; PAI: Polyamide imide; PDMS: Polydimethyl siloxane; PEA: Aromatic polyether amide; PEG: Polyethylene glycol; PEI: Polyether imide; PEO: Polyethylene oxide; PES: Polyether sulfone; PU: Polyurethane; PI: Polyimide; PIM-1: Polymer of intrinsic microporosity; PPO: Polypropylene oxide; PSF: Polysulfone; PVAc: Polyvinyl acetate; PVAm: Polyavinylamine; PVDF: Polyvinylidene fluoride; PVA: Polyvinyl alcohol; PVP: Polyvinyl; SPEEK: Sulfonated aromatic poly (ether-ether-ketone)

Issues and challenges in membrane applications for gas separation: The following issues and challenges occur in membranes application for gas separation:

- Membrane fouling
- High cost
- Concentration of polarization
- Lack of selectivity
- Lack of mechanical resistance
- Sensitivity to chemical attack
- Membrane cleaning
- Module design



(a) Polysulfone



(b) Polyvinyl acetate

Fig. 1: Structure of polysulfone and polyvinyl acetate

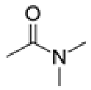


Fig. 2: Structure of Dimethylacetamide (DMAc.)

METHODOLOGY

Materials and membrane fabrication: Polysulfone (PSU) Udel® P-1800 having a glass Transition temperature (T_g) of 185°C was acquired from Solvay Advanced Polymers; L.L.C, U.S. PSU was in minced form. Polyvinyl Acetate (PVAc.) average M_w~100,000 by GPC, beads from Sigma Aldrich having a glass Transition temperature (T_g) 30°C. Dimethyl Acetamide (DMAc.) solvent with a purity of 99.99% was purchased from Merck.

In this process, experimentation on blending of glassy and rubbery polymer that is Polysulfone and Polyvinyl acetate (Fig. 1) is carried out in solvent that is Dimethylacetamide (DMAc) (Fig. 2). The blending is 20% weight/weight. The solvent is 80% and polymer is 20% of total weight. PSU were pre heated during the night to remove any moisture content. Initially PVAc. was allowed dissolving in the DMAc. solvent completely. Then glassy polymer was added. Stirring was continuous for 24 h. Polymers will be dissolving in a solvent at room temperature under continuous stirring to obtain a homogeneous mixture. To obtain a clear solution followed by bath sonication in Transonic Digital S, Elma® for 1 h. for the purpose of degassing. Each polymer be completely dissolved in solvent and there were no signs of agglomeration upon standing the solution Thus we can say that it is a miscible polymer blend. This casting solution was casted on a glass plate by using casting knife with an opening of 200 µm. The

Table 2: Composition of different polymer blend membranes

	Polymer blends	
Membrane sample	Glassy polymer PSF (%)	Rubbery polymer PVAc. (%)
1	100	0
2	0	100
3	95	5
4	90	10
5	85	15
6	80	20

casted membranes were placed in a room temperature for 5 days to evaporate the solvent. For characterization the developed membranes were peeled off from glass plate. The different compositions of polymeric blend membrane are shown in Table 2.

Characterization: The prepared blend membranes were characterized to see the effect of polymer blending on morphology, chemical bonding, thermal stability and Permeability/Selectivity of polymeric blend membranes. For comparative studies pure PSU and pure PVAc. Membranes were also prepared and characterized. Field Emission Scanning Electron Microscopy (FESEM) instrument SUPRA 55 VP by ZEISS was applied to observe the morphology of blend membranes. Surface and cross section microscopic images of developed membranes were taken to observe the blending behavior of polymers by taking specimens from developed membranes. For cross section images membranes samples were dipped in liquid nitrogen and fractured. The surface view magnifications are taken 500 X. The FTIR taken by Perkin Elmer Spectrum one FTIR Spectrometer. The scans are 20 and the wave length range is 4000-450 cm^{-1} and the spectra were obtained from a 200 µm diameter sampling area. The membrane samples were cut at random positions from casting films dried more than 48 h at room temperature and then clamped to the plate. All spectra were corrected for the FTIR characteristic progressive increase in the absorbance at lower wave numbers, using the equipment software. Thermo Gravimetric Analysis (TGA) technique was used to study the thermal stability of polymer blend membranes in the temperature range of 28 to 800°C with heating rate of 10°C/min. The Gas Permeability and selectivity of polymeric blend membrane were analyzed by Carbon dioxide Separation Membrane Unit (CO_2SMU).

RESULTS AND DISCUSSION

FTIR: Thus in this study a complete vibration analysis is attempted and in the present study the FTIR spectra of the PVAc. and PSF were recorded analysis was carried out. Using their results various modes of vibrations and their probable assignment have been discussed. The chemical structures of these chemicals are shown in Fig. 1 and 2 and spectra are shown in following Table 3 to 5.

Table 3: FTIR spectra of pure PSU

PSU spectra	Wave number cm^{-1}
S = O symmetric stretch	1150, 1307
CSO_2C asymmetric stretch	1322
C-O asymmetric stretch	1244, 1260-1000
C_6H_6 ring stretch	1587-1489
OH aliphatic and aromatic stretch	2886, 2938 and 2971

Table 4: FTIR spectra of pure PVAc

PVAc. spectra	Wave number cm^{-1}
C = O stretch	1736, 1760-1670
C-O asymmetric stretch	1244, 1260-1000
C-H phenyl ring substitution band	850, 870-675

Table 5: FTIR spectra of pure DMAc

DMAc spectra	Wave number cm^{-1}
C = O stretch	1736, 1760-1670
C-N stretch	1340-1020

In Fig. 3 the FTIR of polymeric membrane of PVAc. shows the following functional group C = O, C-O and C-H. In pure PVAc. Spectra the wave length of C = O is 1736.00 cm^{-1} and when the membrane is develop in DMAc. solvent, the peak is shifted to 1630.65 cm^{-1} due to the presence of amide group. In the carboxylic group the C-O, the peak is appears from 1244.00 to 1025.72 cm^{-1} due to hydrogen bonding (Ahmed *et al*., 2011). The C-H group in Phenyl ring substitution bends of pure PVAc. is 850.00 cm^{-1}, the band is shifted towards 801.85 cm^{-1} due to asymmetric stretching in CH_3 (Morris, 1943; Jensen *et al*., 1972).

The Fig. 4, the Sulfone group S = O symmetric in polysulfone the wave number is 1150.00 cm^{-1}, the peak is shifted to 995.89 cm^{-1} there is no big change occur due to vibration stretching. The CSO_2C asymmetric stretch the peak is slightly shifted 1323.53 cm^{-1}, due to hydrogen bonding. The C-O asymmetric stretch in PSU the wave number is 1244 cm^{-1}, the peak is shifted to 1018.45 cm^{-1} due to hydrogen shifting bonding (Ahmed *et al*., 2011). The C_6H_6 ring stretch the wave number is 1587.00 cm^{-1} and the peak is on 1579.33 cm^{-1} there is a slightly change in these peaks and remain stable. The OH Aliphatic stretch the wave number is 2886.00 cm^{-1}, band occurs on 2880.15 cm^{-1}, due to weak electro negativity of OH negative ion.

The Fig. 5 to 8, the Sulfone group S = O symmetric in polysulfone the wave number is 1150.00 cm^{-1}, the peak is shifted to 1103.17, 1150.11, 1000.73 and 1095.23 cm^{-1}, respectively there is no big change occur due to vibration stretching. The CSO_2C asymmetric stretch the peak is slightly shifted 1253.39, 1310.26, 1306.41 and 1320.16 cm^{-1}, respectively, due to hydrogen bonding. The C-O asymmetric stretch in PSU and PVAc the wave number is 1244 cm^{-1}, the peak is shifted to 1228.83, 1060.54, 1170.59 and 975.78 cm^{-1}, respectively due to hydrogen shifting bonding (Ahmed *et al*., 2011). The C_6H_6 ring stretch the wave number is 1587.00 cm^{-1} and the peak is on 1580.26, 1580.67, 1566.80 and 1587.27 cm^{-1}, respectively, there is a slightly change in these peaks and remain stable. The OH Aliphatic stretch the wave number is 2886.00, band occurs on 2853.88, 2860.44 2815.93 and 2795.42 cm^{-1}, respectively, due to weak electro negativity of OH negative ion.

The Fig. 5 to 8 the C = O Ketone in PVAc. the peak is shifted to 1732.82, 1730.37, 1709.62 and 1725.46 cm^{-1}, respectively, no change occur in structure bonding

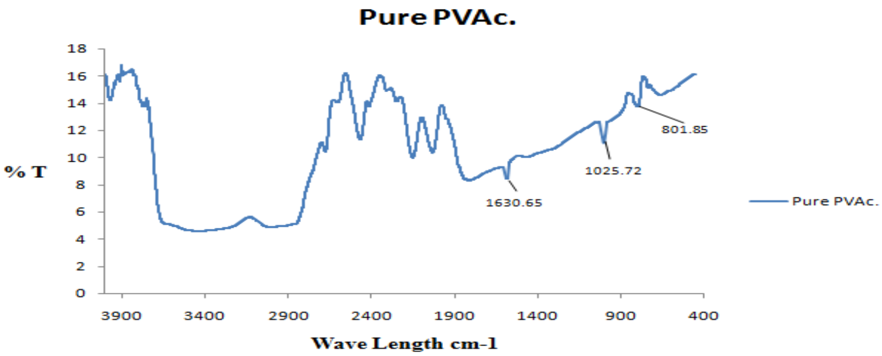


Fig. 3: FTIR of polymeric membrane of PVAc

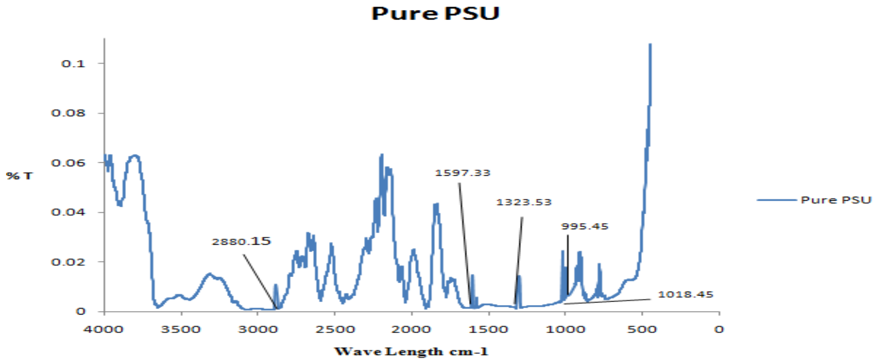


Fig. 4: FTIR of polymeric membrane of PSU

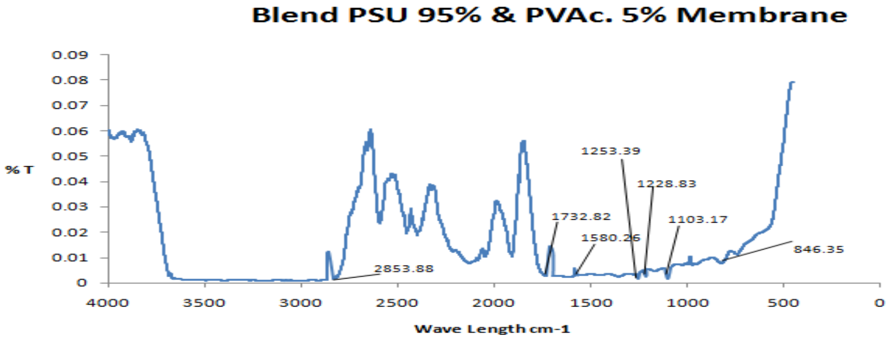


Fig. 5: FTIR of polymeric membrane of PSU 95% and PVAc. 5%

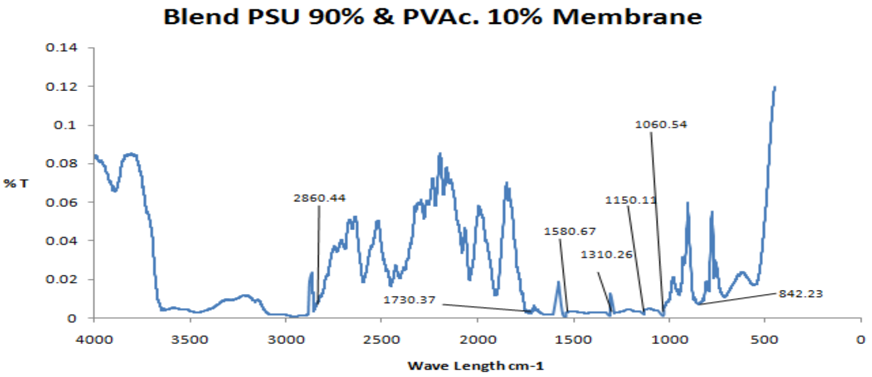


Fig. 6: FTIR of polymeric membrane of PSU 90% and PVAc. 10%

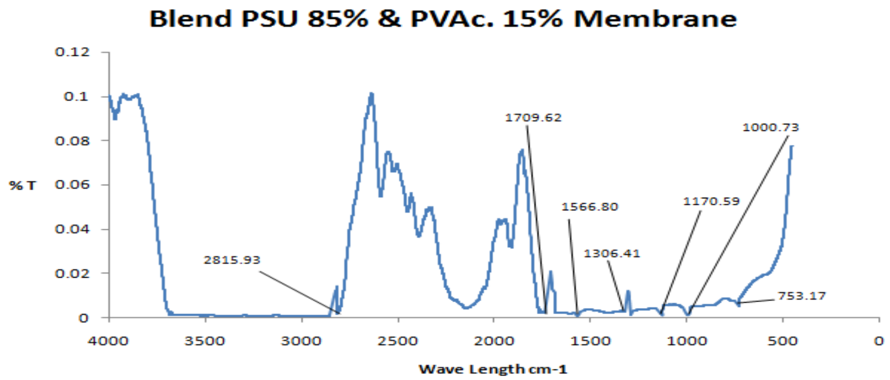


Fig. 7: FTIR of polymeric membrane of PSU 85% and PVAc. 15%

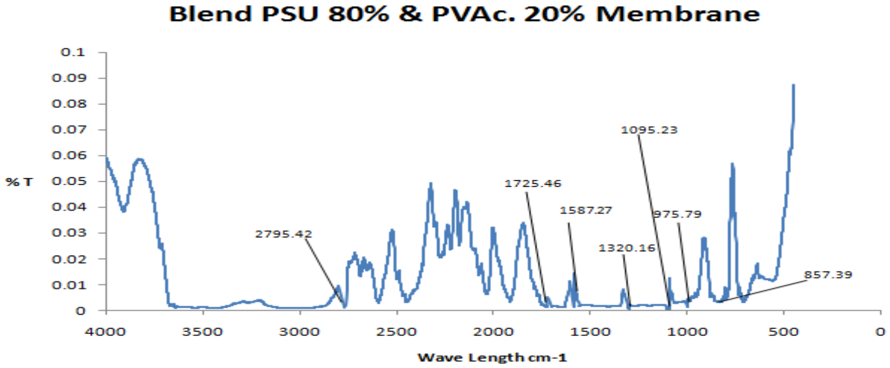


Fig. 8: FTIR of polymeric blend membrane of PSU 80% and PVAc. 20%

but slightly vibration due to stretching. The C-H Phenyl ring substitution band wave number is 850/m, the peak is shifted towards 846.35, 842.23, 753.17 and 857.39 cm^{-1}, respectively, the peak is slightly change from original position but it comes in range due to stable hydrogen bonding.

The miscibility of polymeric blends is confirmed from FTIR study because a few shifts are seen in FTIR spectra. FTIR analysis of this blended membrane showed that the frequency was shifted for the characteristic peaks involving di-aryl sulfone, C = O, C-O groups of PSU/PVAc. These spectral changes indicated the existence of molecular interaction among the polymeric blends; highlight the compatible nature among each other.

TGA: The thermal stability of polymeric blend membranes was analyzed by Thermal Gravimetric Analysis. Fig. 9 represents the TGA graph of blend membranes. Pure PSU, PVAc. and blend membrane were synthesized in DMAc. solvent. Polysulfone Polymer started degradation at 535.13ºC and thermal degradation continues till 570.38ºC and PVAc. membrane started degradation at 318.54ºC and final degradation temperature is occurring at 355.38ºC.

The blend membranes have shown a degradation behavior in between the pure polymers. Addition of PVAc in PSU has change the thermal stability of polymeric blend membranes. The 95% PSU and 5% of PVAc. the stability of resultant blend membrane is change from 535.13 to 532.48°C due to addition of 5% PVAc. Whereas an addition of 10 % PVAc. in 90% PSU

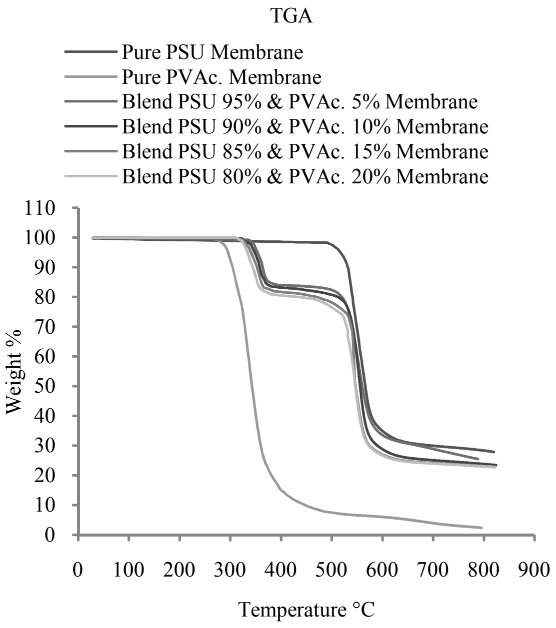


Fig. 9: TGA graph of developed membranes

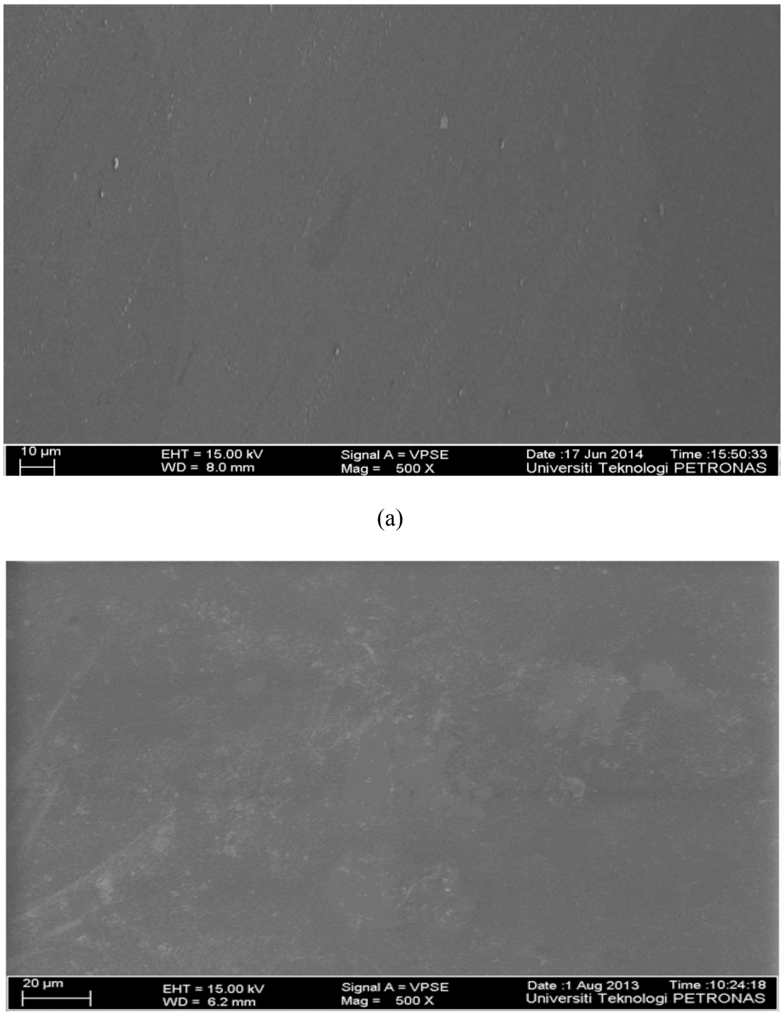
Table 6: Thermal degradation temperature of polymeric blend membrane

Membrane sample	Polymer blends PSU (%)	 PVAc. (%)	Degradation onset temperature (°C)	Maximum degradation temperature (°C)
1	100	0	535.13	570.38
2	0	100	318.54	355.38
3	95	5	532.48	566.17
4	90	10	530.16	561.24
5	85	15	528.87	556.39
6	80	20	527.38	553.92

has change the thermal degradation onset temperature to 530.16°C and degradation end temperature is observed to be decreased to 561.24°C. The 85% PSU and 15% PVAc. Membrane degradation temperature occur at 528.87°C. The membrane of 80% PSU and 20% PVAc. the degradation temperature further reduces to 527.38°C and its max. Degradation temperature arises at 553.92°C. Increase the PVAc. Degradation temperature were fall as an increase in the concentration of PVAc. in PSU. In the previous studies thermal degradation results are in agreement (Rafiq *et al*., 2012). The thermal Degradation temperatures are shown in Table 6.

FESEM: The flat sheet dense membranes of PSU/PVAc. blend was synthesized by solution casting and evaporation method. The FESEM images of membranes are shown in Fig. 10 and 11. The morphology of blend membrane is compared with pure PSU and PVAc. Membranes. In Fig. 10a and b surface of pure PSU and PVAc. membranes are shown. It is clear from these images that dense membranes are synthesized effectively. There were no pores that can be seen on the surface. Dense structure of PSU and PVAc. are confirmed from these images. In figure 10c PSU 95%/PVAc. 5% and 10d PSU 90%/PVAc. 10% surfaces of blend membranes are uniform with no pores and no phase separation which confirms the miscibility of both polymers (Houde *et al*., 1992). In Fig. 10e PSU 85%/PVAc. 15% and Fig. 10f PSU 80% and PVAc. 20%, the surface of these membranes are also dense.

The cross section of pure PSU and pure PVAc., polymeric blend membranes were also shown a dense structure in Fig. 11a and b. In Fig. 11c to f PSU/PVAc. blend membrane are presenting the highly packed uniform pores structure but the size of the pores are



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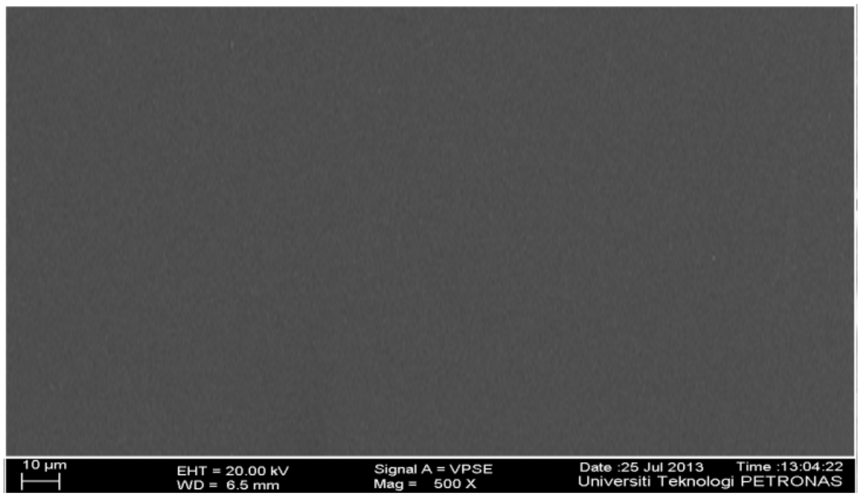
(b)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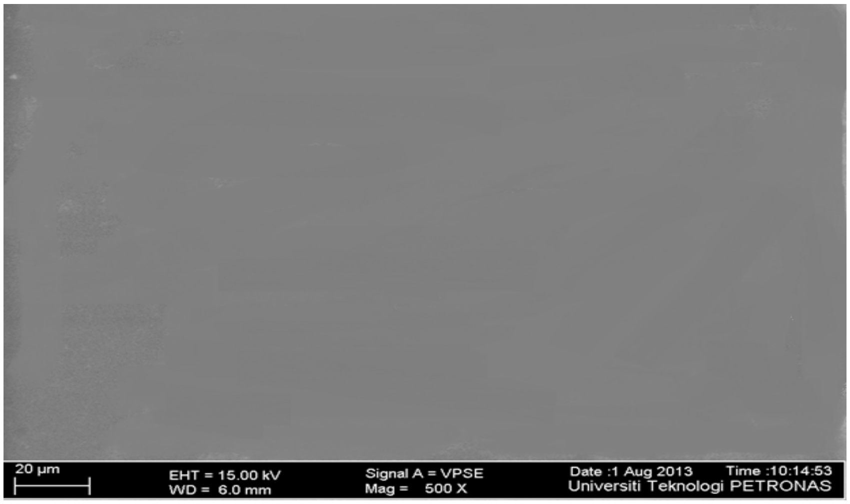
(c)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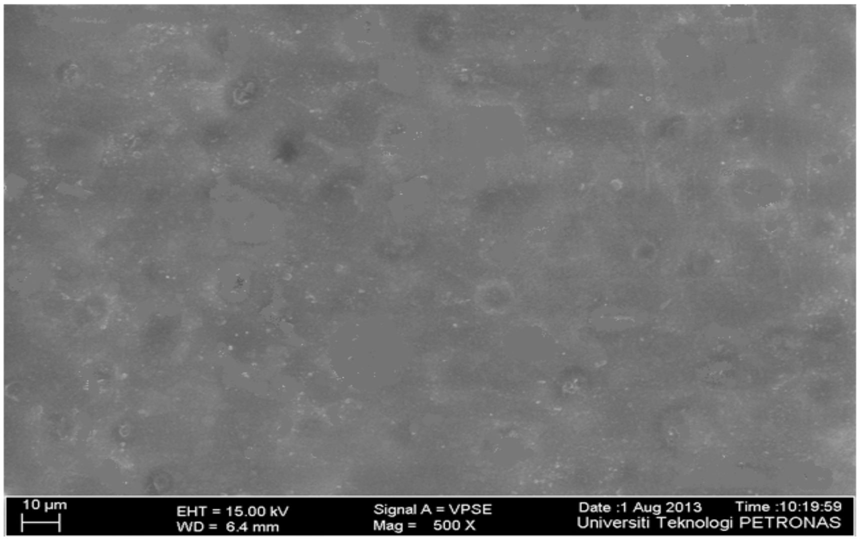
(d)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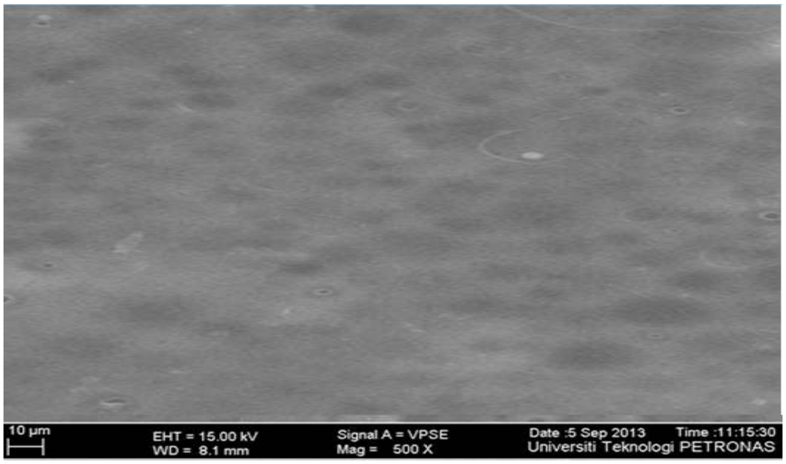
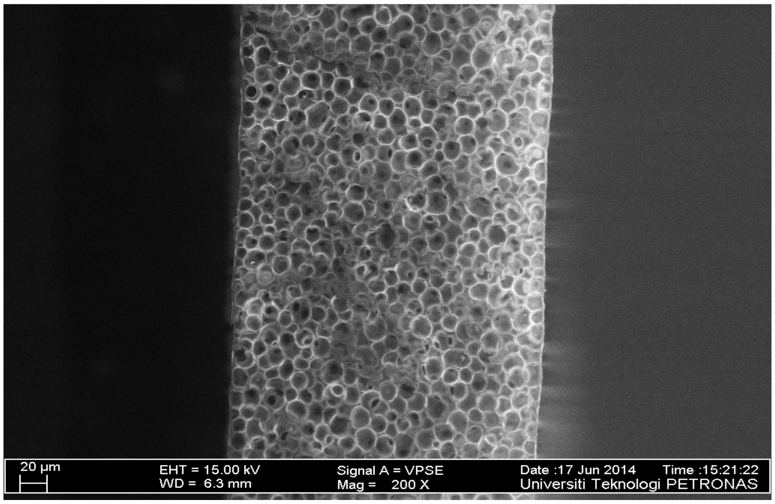
(e)

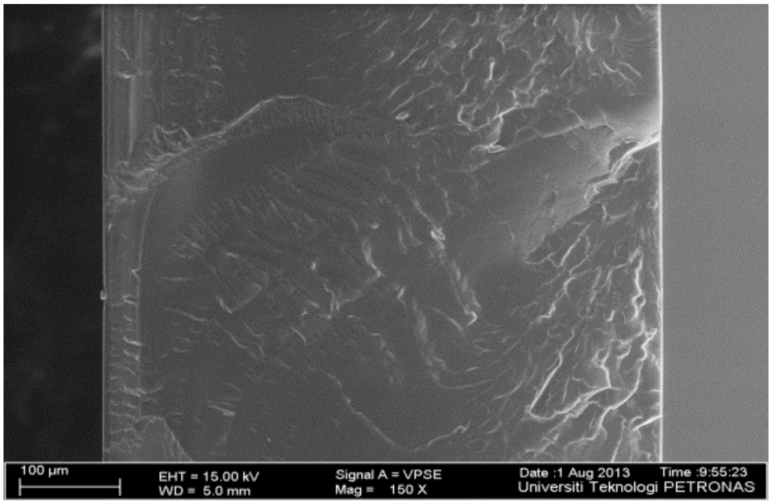


(f)

Fig. 10: Membrane surface image of (a) pure PSU, (b) pure PVAc., (c) PSU 95%/PVAc. 5%, (d) PSU 90%/PVAc. 10%, (e) PSU 85%/PVAc. 15% and (f) PSU 80%/PVAc. 20%



(a)



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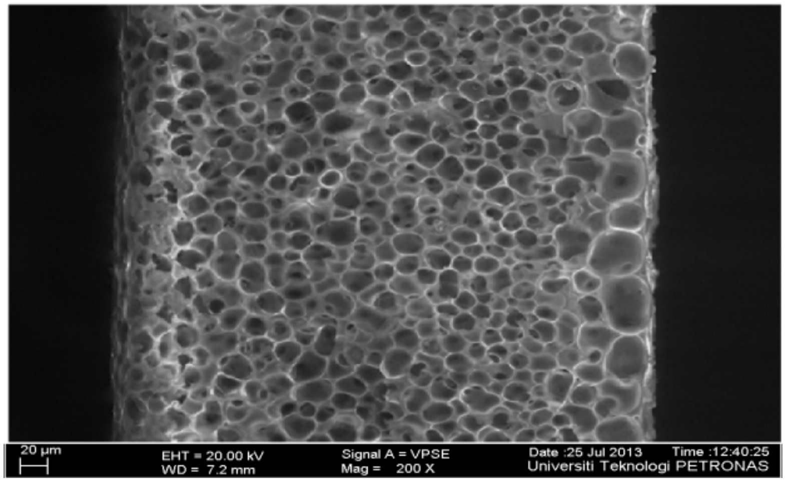
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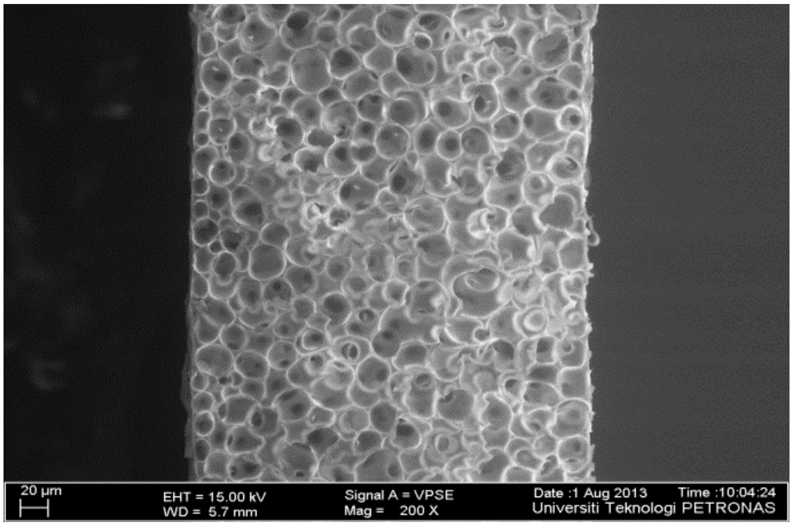
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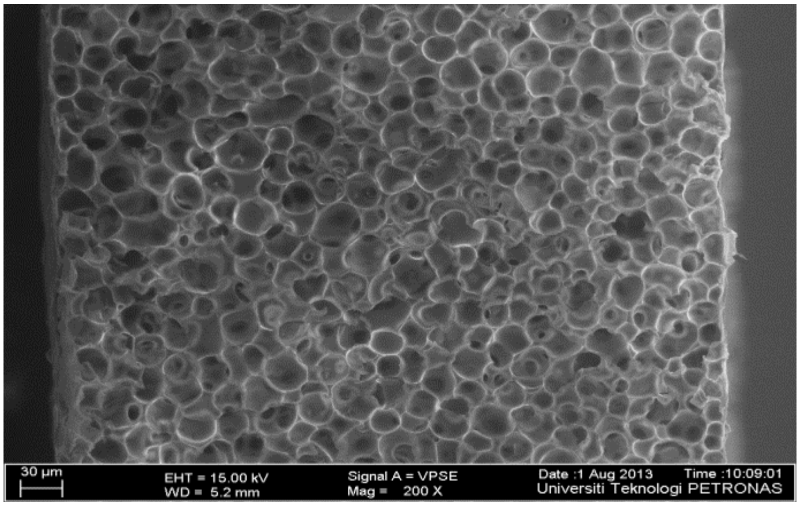
(d)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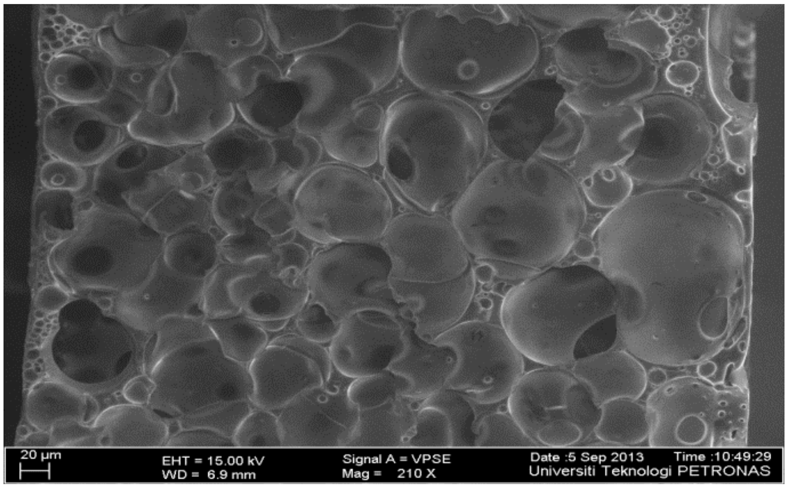
(e)



(f)

Fig. 11: Membrane cross section view of (a) pure PSU, (b) pure PVAc., (c) PSU 95%/PVAc. 5%, (d) PSU 90%/PVAc. 10%, (e) PSU 85%/PVAc. 15% and (f) PSU 80%/PVAc. 20%

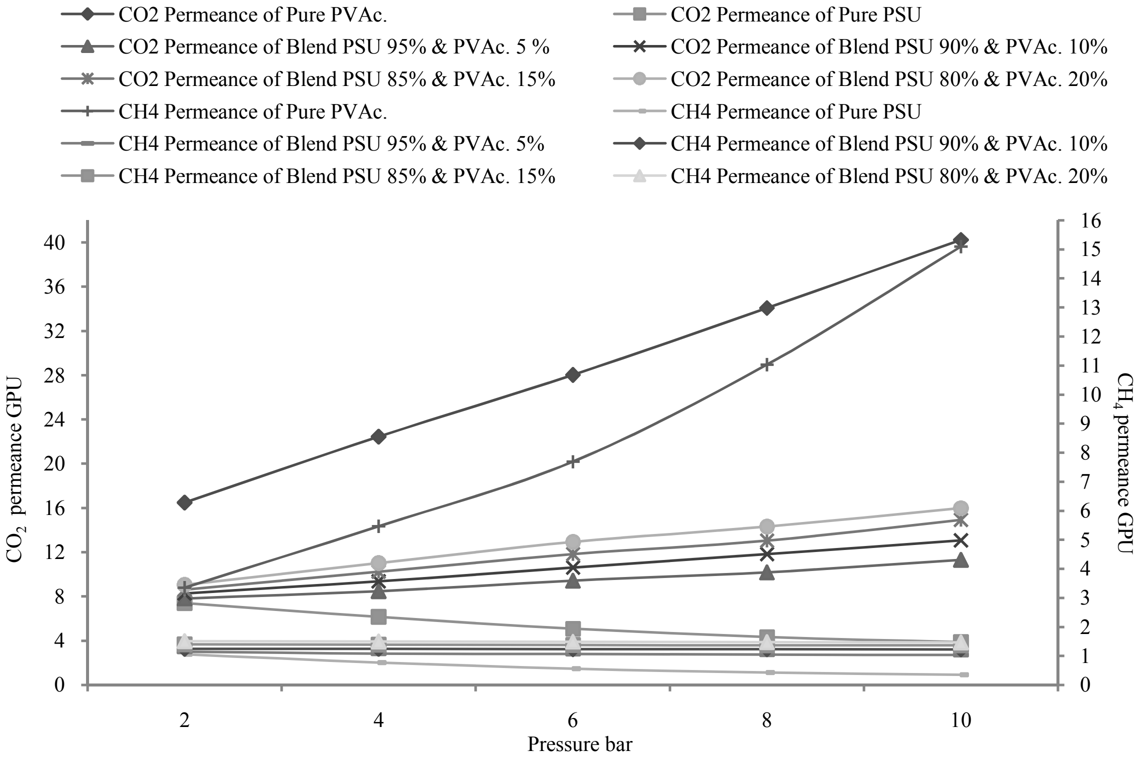


Fig. 12: CO_2 and CH_4 permeance of polymeric blend membrane in various feed pressures

different. The cross section indicating the good interaction between these polymers.

Gas permeability: In industrial application CO_2 needs to be separated from the CH_4. To evaluate the viability of the PSU/PVAc. membranes for realistic separation of CO_2 from CH_4, the performance of this reactive polymeric blend membrane for CO_2/CH_4 mixture separation were investigated. The permeance of CO_2/CH_4 mixture was measured at different operating pressures. The apparatus used for gas mixture permeation experiment is CO_2SMU. The flow rate of CO_2/CH_4 is 0.1 m^3/sec were controlled by flow meters/controllers. The experiments were carried out at room temperature 298+2 K and environmental pressure of 101+2 kPa.

Table 7: CO_2 permeability of polymeric blend membrane

Pressure ‘bar’	CO_2 Permeance ‘GPU’ Pure PVAc.	Pure PSU	Membrane PSU 95% and PVAc. 5%	Membrane PSU 90% and PVAc. 10%	Membrane PSU 85% and PVAc. 15%	Membrane PSU 80% and PVAc. 20%
2	16.48457	7.406534	7.81009912	8.27324001	8.8474467	9.31269
4	22.42997	6.153237	9.64533443	10.4357516	10.9109284	11.759
6	28.01494	5.090528	11.8132434	12.4455311	13.4363792	14.1209
8	34.08332	4.342335	13.9700672	14.7860296	16.2477955	16.8858
10	40.24454	3.870025	16.956511	17.524489	18.8391543	19.7426

Table 8: CH_4 permeability of polymeric blend membrane

Pressure ‘bar’	CH_4 Permeance ‘GPU’ Pure PVAc.	Pure PSU	Membrane PSU 95% and PVAc. 5%	Membrane PSU 90% and PVAc. 10%	Membrane PSU 85% and PVAc. 15%	Membrane PSU 80% and PVAc. 20%
2	3.347096	1.05458	1.14238501	1.24303766	1.3935492	1.50553
4	5.458785	0.76509	1.23038219	1.36701393	1.47284479	1.59879
6	7.692395	0.56126	1.33476803	1.44539563	1.57661209	1.68482
8	11.029780	0.42655	1.40475883	1.52708411	1.69670509	1.79365
10	15.091700	0.34663	1.55121334	1.64252683	1.78661018	1.8893

Table 9: Selectivity CO_2/CH_4 of polymeric blend membrane

Pressure ‘bar’	Selectivity CO_2/CH_4 Pure PVAc.	Pure PSU	Membrane PSU 95% and PVAc. 5%	Membrane PSU 90% and PVAc. 10%	Membrane PSU 85% and PVAc. 15%	Membrane PSU 80% and PVAc. 20%
2	4.925036	7.023182	6.83666	6.65566	6.181176	6.00655
4	4.108967	8.042537	7.83183	7.56089	7.411650	7.35579
6	3.641901	9.069813	8.85820	8.61197	8.521789	8.48848
8	3.090119	10.180110	9.76871	9.61837	9.547945	9.41318
10	2.666667	11.164740	10.92620	10.65470	10.550000	10.45120

According to Houde *et al.* (1992), pure PSU polymer shows different CO_2 permeation characteristics. The observed gas permeability’s, structural, physical compatibility and properties of PSU, PVAc. Blends make the homogenous blend membranes quite attractive. Compared to pure PSU membrane, they exhibit improve the CO_2 permeability due to the presence of PVAc. in the blend. Compared to pure PVAc. Membrane, they exhibit improve the selectivity due to the presence of PSU in the blend. Gas permeability study of PSU/PVAc. and blend membranes were evaluated using pure gas CO_2 and CH_4 at five different pressure 2, 4, 6, 8 and 10 bars.

Figure 12 relates the Table 7 and 8, the permeance of CO_2 and CH_4 of pure PSU membrane is reduces with the increase of pressure. On the other hand pure PVAc. membrane the permeance of CO_2 and CH_4 is increases with the increase of pressure. As the percentage of PVAc. an increase in PSU, the permeability of CO_2 is increases with the increases of pressure where as permeability of CH_4 is also slightly increases, when the pressure increases.

Figure 13 relates the Table 9; the selectivity of CO_2/CH_4 of pure PSU is increases with the increase of pressure. On the other hand pure PVAc. membrane, the selectivity of CO_2/CH_4 decreasing with increasing the pressure. When the blend membranes were synthesized, as percentage of PVAc. increases in PSU, the selectivity increases with the increases of pressure.

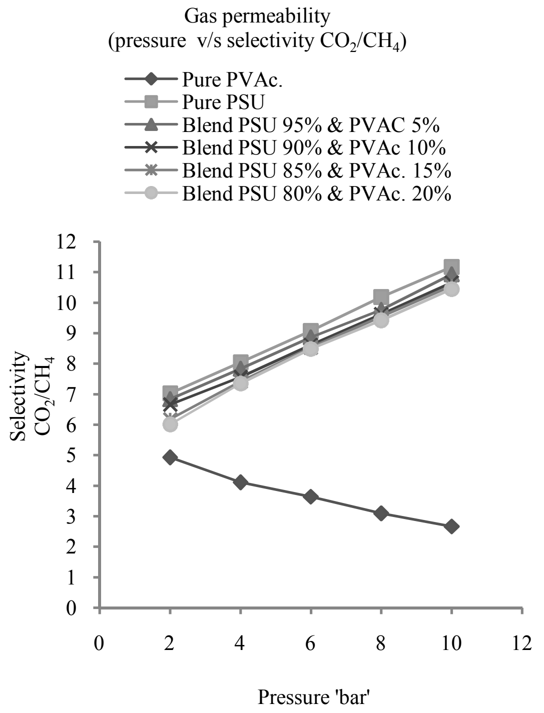


Fig. 13: CO_2/CH_4 selectivity’s of polymeric blend membrane in various feed pressures

It is concluded that the pure PSU glassy polymeric membrane and pure PVAc. Rubbery membrane have completely miscible the properties to increase the blend membrane properties that is, the permeance of CO_2 is increases with the increases of pressure and selectivity is also increases. Therefore, PSU/PVAc. Polymer blends can be viewed as new, economical, high performance raw material suitable for the preparation of gas separation membrane to removes CO_2 from CH_4 with enhanced permeation and selectivity.

CONCLUSION

Polymer blend membrane is an advanced technique for gas separation, CO_2 from CH_4. The morphology of the blend membranes is homogenous and no micro voids are seen on surface and cross sections are symmetric porous. FTIR analysis also confirmed that the polymer blends are completely miscible among each other. Moreover there is no cross linking or formation of intermediates. These spectral changes indicated the existence of molecular interaction among the polymeric blends; highlight the compatible nature with each other.

The permeance of CO_2 is increasing with the increasing of the pressure in the PSU/PVAc. blend membranes. PSU/PVAc. flat sheet membrane exhibit gas permeability coefficient which vary monotonically between the values of the two pure polymers. In the future work we also add the inorganic fillers like carbon molecular sieves; zeolites in blend which is further enhanced the polymeric blend membrane. Therefore, this can increase the economic process in gas trade.

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